TRANSLATION

UNION OF SOVIET SOCIALIST REPUBLICS

USSR State Committee

On Matter of Inventions and Discoveries

DESCRIPTION OF INVENTION

For Inventor's Certificate of SU 1810382 A1

Int. Cl.[5]:	C 10 M 137/04
Application No.:	4883424/04
Application Date:	August 1, 1990
Publication Date:	April 23, 1993 Bulletin No. 15

LUBRICANT ADDITIVE

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Applicant:

Institute of Physical Chemistry, Academy of The Sciences of the USSR

References:

GOST [All-Union State Standard] 14625-69

U.S. Patent 4,169,800
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Summary

Essence of Invention: A lubricant additive containing a mixture of alkylfluroalkyl phosphates of the formula

$$[H(CF_2CF_2)_nCH_2O]_2P(O)(OR)\ I.\quad [H(CF_2CF_2)_nCH_2O]P(O)(OR)_2\ II.$$
$$[H(CF_2CF_2)_nCH_2O]P(O)(OR)(OH)\ III.$$

where R=C_{12}-C_{16} and n=2-4, in the following ratio of components, %: I 10-75, II 20-87, III 1-5. 2 Tables.

The invention relates to lubricants that are characterized by an additive containing a mixture of organic phosphorus- and halogen-containing compounds, which impart anticorrosion, antiwear and antifriction properties when added to oil and lubricants. Such additives can be used in the machine construction and in transport as an additive to industrial motor and transmission oils.

The goal of the present invention is an improvement of the anticorrosion, antiwear and antiseizing properties of lubricant oils and also an improvement of their deemulsifying properties.

This goal is achieved by the addition to the oil of an alkylfluroalkyl phosphate additive that is a mixture of phosphoric acid esters of the formula:

$$I\ [H(CF_2CF_2)_nCH_2O]_2P(O)(OR)$$
$$II\ [H(CF_2CF_2)_nCH_2O]P(O)(OR)_2$$
$$III\ [H(CF_2CF_2)_nCH_2O]P(O)(OR)(OH)$$

where R=C_{12}-C_{16},

n=2-4,

in a component ratio, % by weight of 10-75: 20-87: 1-5.

This mixture can be obtained by the synthesis, under specially selected reaction conditions, of fluorine-containing alcohol telomers with phosphorus oxychloride and subsequent partial hydrolysis of the products.

From the test results given in Table II, one can conclude that each of the components of the inhibitor taken separately (Examples 3, 6, 9, 10, 12) exhibits poor antiwear and anticorrosion properties and has lower deemulsifying capacity by comparison with the disclosed mixture. The mixture containing partial hydrolyzed products with an upper limit more than 5 [%] for the third component (Example 4) also develops poorer anticorrosion, antiwear and deemulsifying properties than mixtures proposed for the invention.

Below are examples of the preparation of the disclosed substances.

Example 1 (see Table I)

We add a solution of alcohol telomer, n=2, of the formula $H(CF_2CF_2)_2CH_2OH$ to a solution of freshly distilled phosphorus oxychloride in a stream of inert gas with stirring at 0°C over a period of 40 min so that the temperature of the reaction mixture does not rise above 2°C. At the end of the addition, the mixture is stirred for another 2-3 h, with the completion of the reaction being verified from the disappearance of ν_{OH}=3620 cm^{-1} in the IR spectrum of the reaction mixture. Then we add a solution of dodecyl alcohol (0.2 M) at room temperature over a period of 0.5 h and 0.31 M of a saturated solution of alkali. The sodium chloride precipitate is filtered out, the solvent (benzene) is evaporated out of the mother solution. The residue is viscous liquid that crystallizes upon standing and has m.p. 50-52°C. The yield is 98%. IR spectrum (CCl_4 cm^{-1});: ν_{P-OH}=2700, $\nu_{P=O}$=1210, ν_{C-F}=1020-1040, 1180-1200. According to ^{31}P NMR data, the composition of the mixture of components I, II, III corresponds to 10, 87, 3%.

Example 13

A solution of alcohol telomer, n=4, of the formula $H(CF_2CF_2)CH_2OH$, 0.1 M in 60 ml abs. benzene, is added over a period of 1 h to a solution of freshly distilled phosphorus

oxychloride (0.1 M in 50 ml abs. benzene) with stirring and in a stream inert gas at -5°C. At the end of the addition, the mixture was stirred for 3-3.5 hours until the ν_{OH}=3620 cm^{-1} band in the IR spectrum of the reaction mixture disappeared. Then a solution of 0.2 M hexadecyl alcohol in 70 ml abs. benzene is added at room temperature over a period of 0.5 h and then immediately 0.31 M saturated solution of alcohol is added. The precipitate is filtered out. The solvent is evaporated out of the mother solution. The remainder is a white fusible substance, m.p. 70-71°C. The yield is 99%. The IR spectrum is identical to that of Example 1. According to the ^{31}P NMR data, the composition of the mixture of components I, II, and III corresponds to 75, 20, 5%.

Corrosion tests of the synthesized substances were carried out by the techniques of GOST 9054.75, procedures 3 (Immersion Into Sea Water) and 5 (Displacement of the Corrosive Electrolyte HBr). The degree of protection was determined from the damaged surface area on steel 45, in %.

The determination of the deemulsifying capacity was done by the procedure of GOST 12068-66 by determining the time and percent of stratification of an emulsion when the additives are introduced in the amount of 0.5-1% of the disclosed substances to T-46/1-2% SIM transformer oil.

The characteristics of friction and wear were measured on a Philips machine with complete submersion into the oil, at a load of 20 kg, slip velocity of 0.06 m/sec, test time 1 h. The friction pair consisted of spheres of SHKH-15 steel 8 mm in diameter.

The test results are given in Table II.

As can be seen from the experimental data (Table II), the best protective properties and ability to reduce wear are provided by compositions containing components I, II, and III in ranges of 10-75%, 20-87%, 1-5%. Specifically compositions 1, 2, 5, 8, 11, and 13.

Claims

A lubricant additive that contains alkyl phosphate compounds, which is distinguished by the fact that, with the goal of improving anticorrosion, antiwear, antiseizing and deemulsifying properties, the additive contains as the alkyl phosphate compounds a mixture of alkylfluroalkyl phosphates of the general formulas:

$$[H(CF_2CF_2)_nCH_2O]_2\ P(O)\ (OR): (I).$$
$$[H(CF_2CF_2)_nCH_2O]\ P(O)\ (RO)_2: (II).$$
$$[H(CF_2CF_2)_nCH_2O]\ P(O)\ (OR)\ (OH): (III)$$

where $R=C_{12}-C_{16}$,

$n=2-4$

in the following ratio of components, % by weight: compound of general formula I 10-75: compound of general formula II 20-87: compound of general formula III 1-5.

Table I. Examples of disclosed substances and their composition according to ^{32}P NMR data

Composition of mixture	$n=2, R=C_{12}H_{25}$			$n=2, R=C_{16}H_{33}$			$n=4, r=C_{12}H_{25}$			$n=4, R=C_{16}H_{33}$			
	Example			Example			Example			Example			
	1	2	3	4	5	6	7	8	9	10	11	12	13
I	10	67	100	30.62	75	0	12.37	48.99	0	96.94	31.63	0	75
II	87	30.93	0	59.18	24	100	83.50	48.50	100	0	63.27	0	20
III	3	2.07	0	10.20	1	0	4.13	3.02	0	3.06	5.10	100	5

Table II. Results of tests of disclosed substances

Ne/Ne	Example	Corrosion			Seizure P_k, kg	Wear, mm	Coefficient of friction	Deemulsifying properties, %
		Concentration, %	Degree of damage in sea water*	Displacement of HBr**				
1	1	1	0	0	98	0.38	0.100	96
2	2	1	0.5	0	100	0.39	0.110	100
3	3	1	15	0	80	0.45	0.116	90
4	4	1	11	0	79	0.40	0.112	91
5	5	1	0	0	100	0.39	0.100	100
6	6	1	10-14	0	79	0.42	0.130	95
7	7	1	0	0	96	0.36	0.100	95
8	8	1	1	0	100	0.38	0.111	100
9	9	1	20-25	0	75	0.45	0.120	86
10	10	1	5-6	0	90	0.37	.0111	100
11	11	1	0	0	98	0.38	0.104	96
12	12	1	25-30	0	78	0.42	0.115	89
13	13	1	0	0	101	0.38	0.100	95
14	Oil ASV-5	1	100		50	0.78	0.131	0
15	DF-11	1	100		72	0.52	0.141	–
16	SIM	1	30	40		1.85	0.120	–
17	Proxamin	1	–	–	–	–	–	81
18	Inhibitor according to prototype	–	5-7	3-5	63 —	0.69	0.130	40

* Test duration 20 h.
** Test time 4 h.
*** Inhibitor according to prototype – a mixture of mono- and dialkyl phosphates (Alk-C_8-C_{12}).

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